

Advanced Processing of the Optical Surface on Large Lightweight Mirrors

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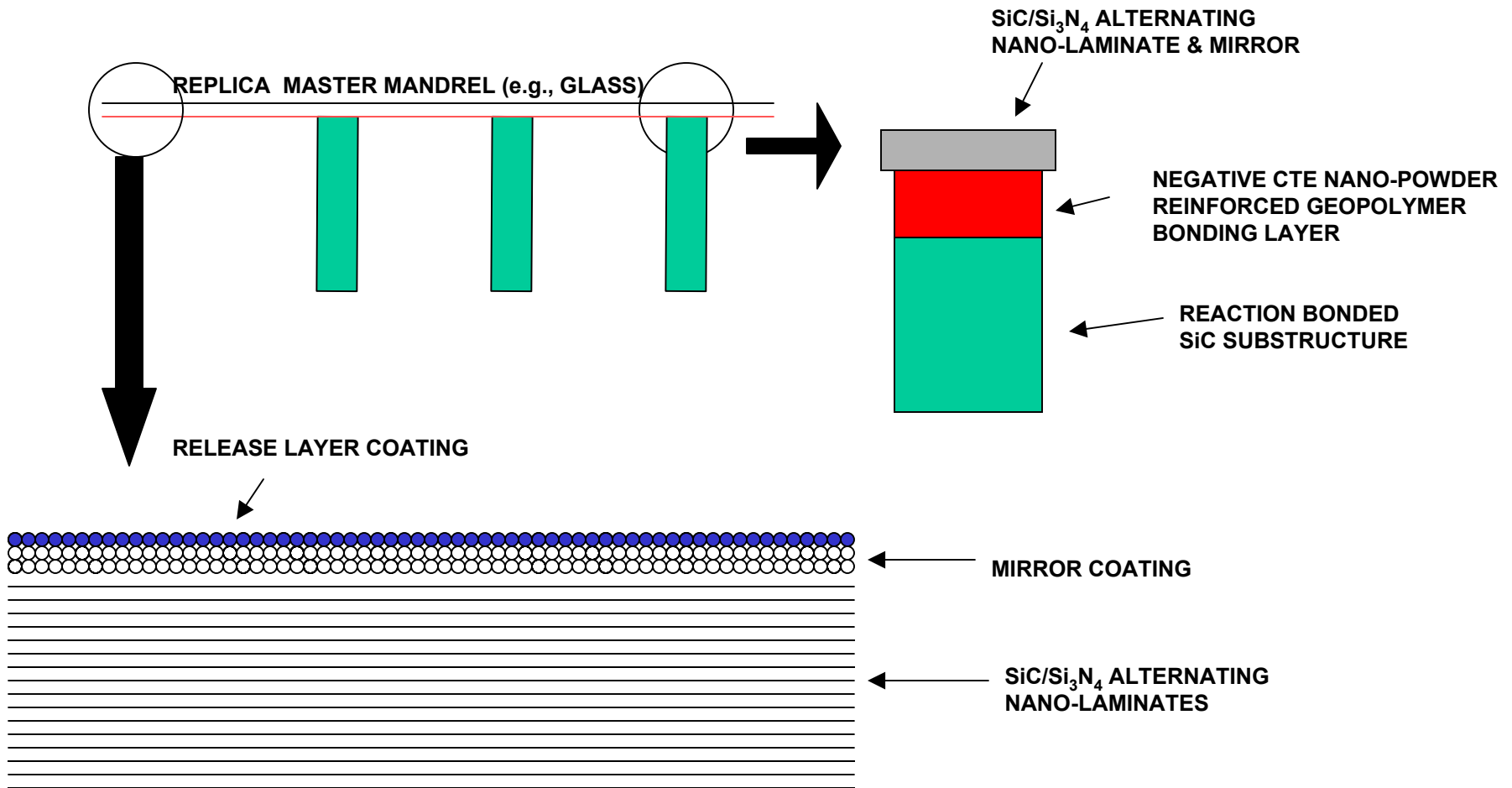
OUTLINE

- * Objectives**
- * Technical Approach (Replication Technique)**
 - SiC/Si₃N₄ Nano-Laminate**
 - Negative CTE Powder/CDD Processed Composite**
- * Critical Issues**
- * Preliminary Experimental Results**
 - Nano-Laminate Deposition**
 - Geopolymerization**
 - Negative CTE Nano-Powder Synthesis**
 - Chemically Driven Densification (CDD)**
- * Summary and Future Work for Next Few Months**

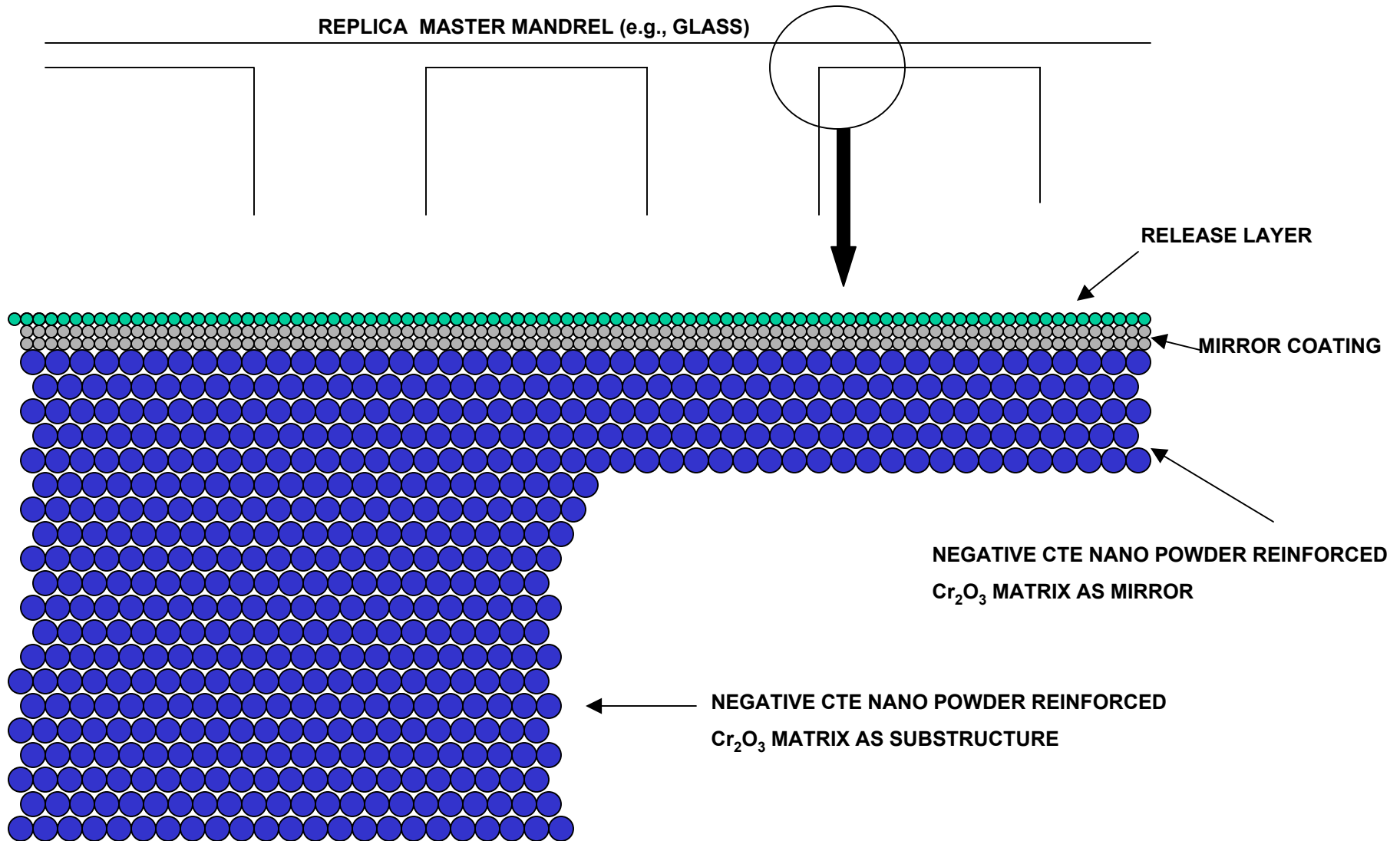
OBJECTIVES

- * Deposition Processing Optimization:
SiC/Si₃N₄ Alternating Nano-Laminate
Release Layer Coating/Concept**
- * Production of –CTE Nano-Powder in large Quantity**
- * -CTE Powder/Geopolymer Composite Fabrication
and Bond Strength measurement of SiC/SiC**
- * -CTE Nano-Powder/CDD Composite Fabrication**

Schematic Drawing of SiC/Si₃N₄ Nano-Laminate Mirror Assembly Concept



Schematic Drawing of - CTE Nano-Powder Reinforced CDD Processed Uni-Body Composite Mirror Assembly Concept



CRITICAL ISSUES

- * Release Layer Coating/Concept :**

 - Easy Release from Master Mandrel without Distortion**

- * SiC/Si₃N₄ Alternating Nano-Laminate :**

 - Microstructural Uniformity within the Layer**

 - Mechanical Strength of the Laminate vs. # of Layers**

- * -CTE Powder/Geopolymer Composite Bond :**

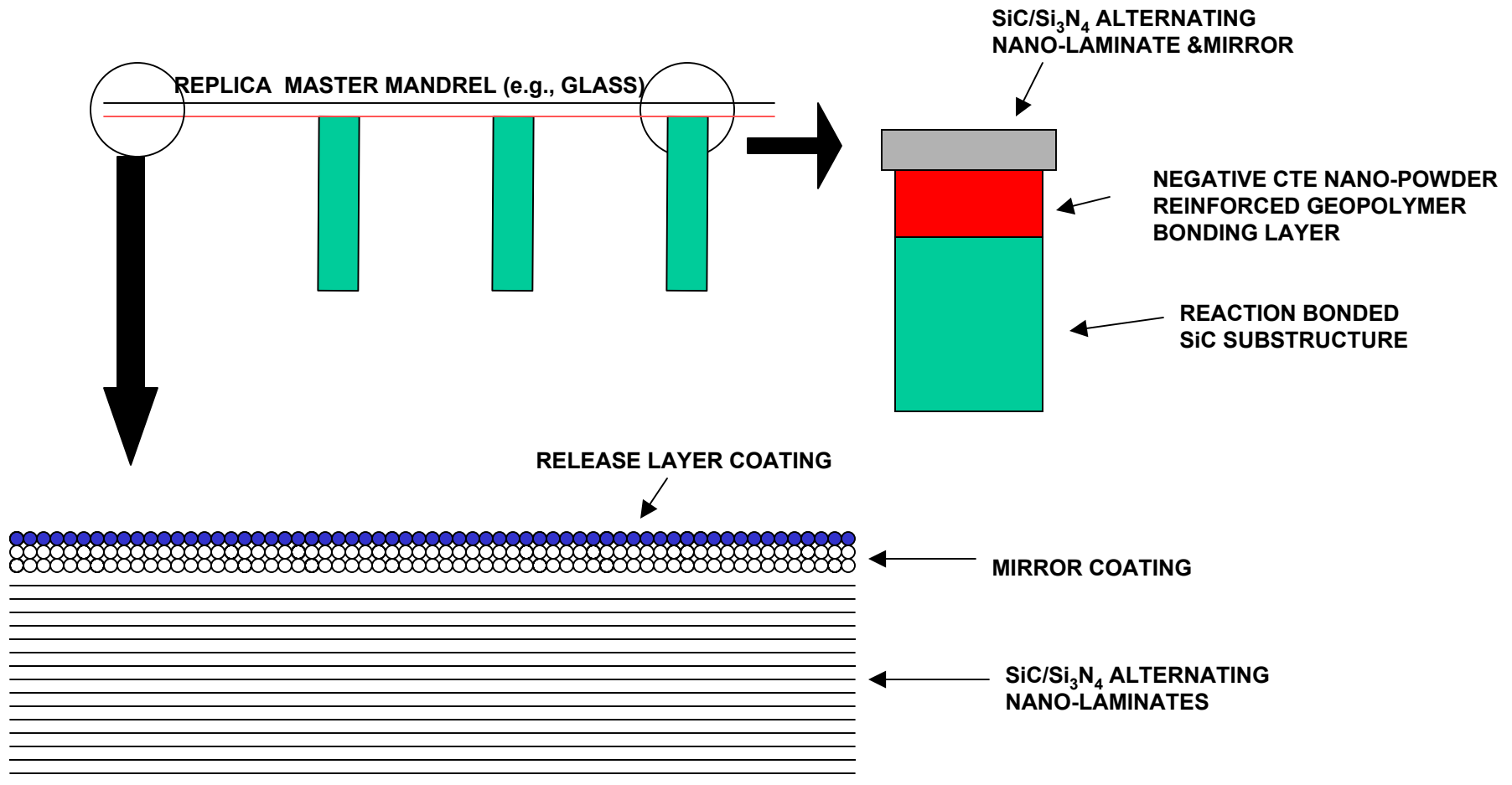
 - Adequate Bond Strength for SiC/SiC Joint**

- * -CTE Powder/CDD Composite Fabrication**

 - Large Negative CTE Nano-Powder production**

 - Exothermic Reaction Control (-CTE powder, Mandrel, etc.)**

Schematic Drawing of SiC/Si₃N₄ Nano-Laminate Mirror Assembly Concept



EXPERIMENTAL RESULTS

Single Component Magnetron Sputtering Deposition

SiC : Deposition Optimization (7 trials) by Varying Argon Flow Rate and Chamber Pressure and Deposition Time and Distance from Target to Si Substrate

Si₃N₄ : Deposition Parameter Optimization Started

Fugitive Release Layer

KBr : Thermal Evaporation Deposition

SiO₂ : Thermally Grown on Si

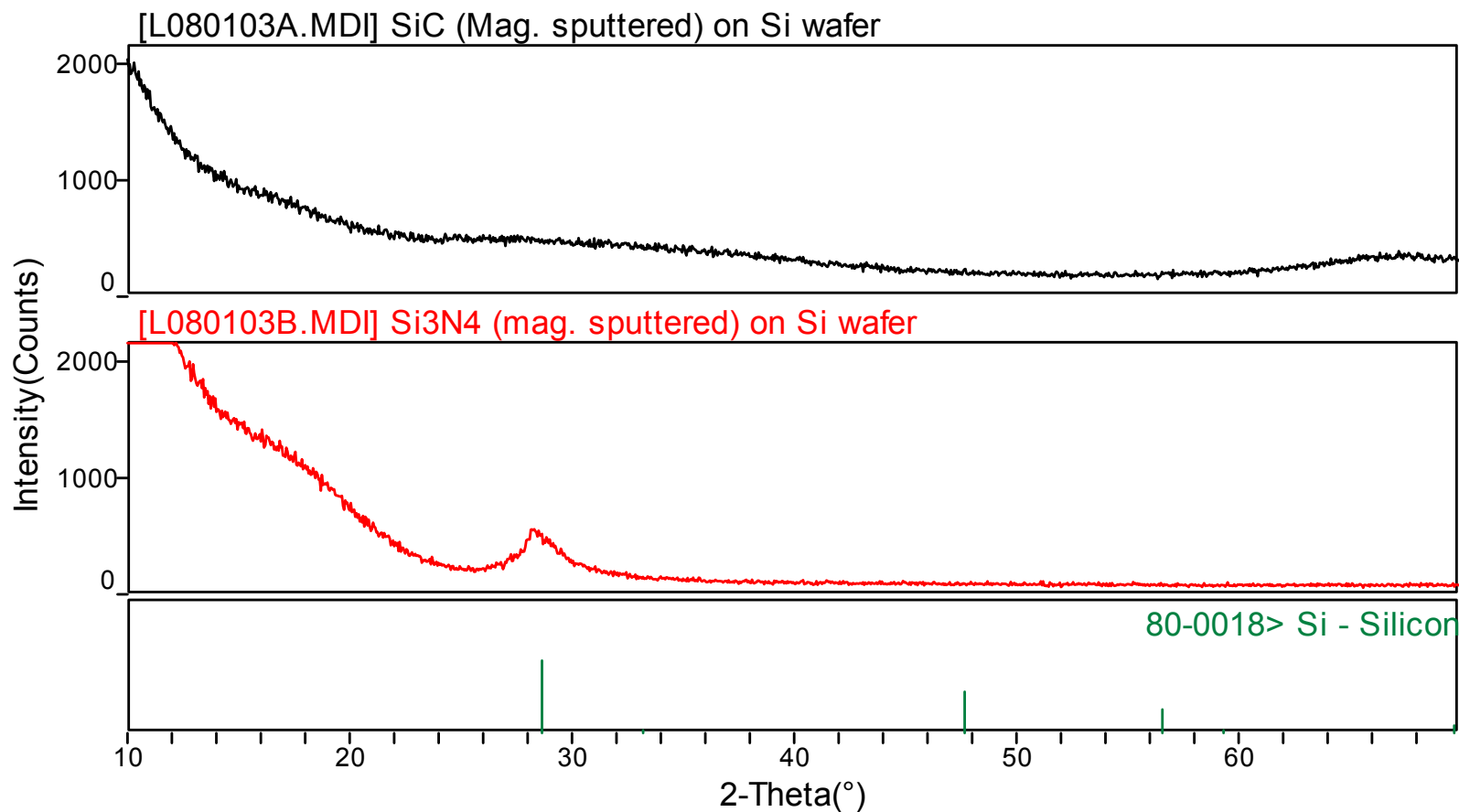
SiC/Si₃N₄ Alternating Multi-Layer Deposition

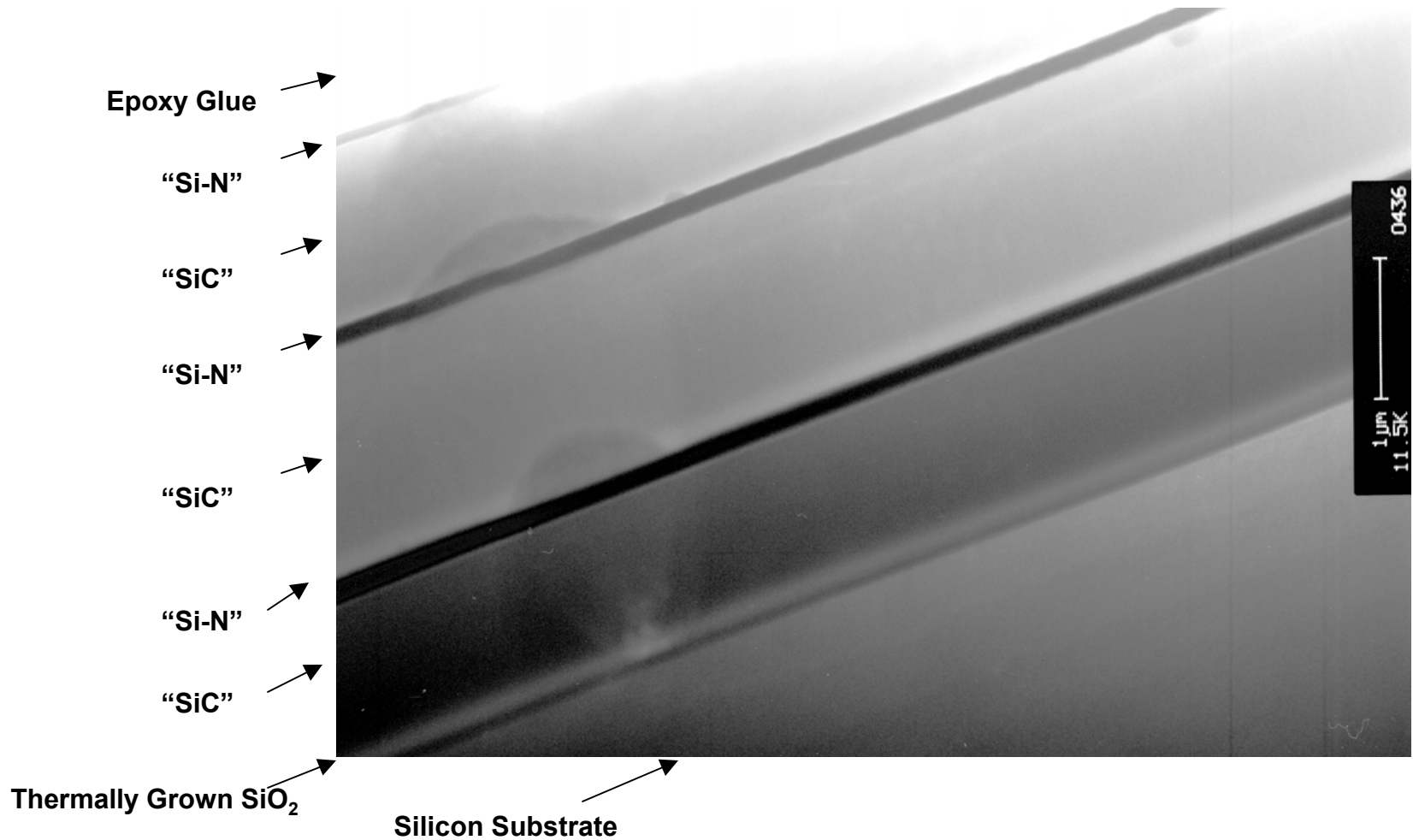
7 Alternating Layers on with and without SiO₂ Release layer

XRD and Cross-sectional SEM/TEM micrographs

XRD on Single components revealed Amorphous

XRD Patterns of SiC and Si₃N₄ Coatings on Si Wafer Showing Amorphous nature of the Coatings





**Cross-Sectional TEM Micrograph of Multi-Layer Coating
(The Compositions of the Coating Layers are Tentative)**

Potential negative CTE oxides

(CTE Range = $-5 \sim -9 \times 10^{-6}/\text{K}$)

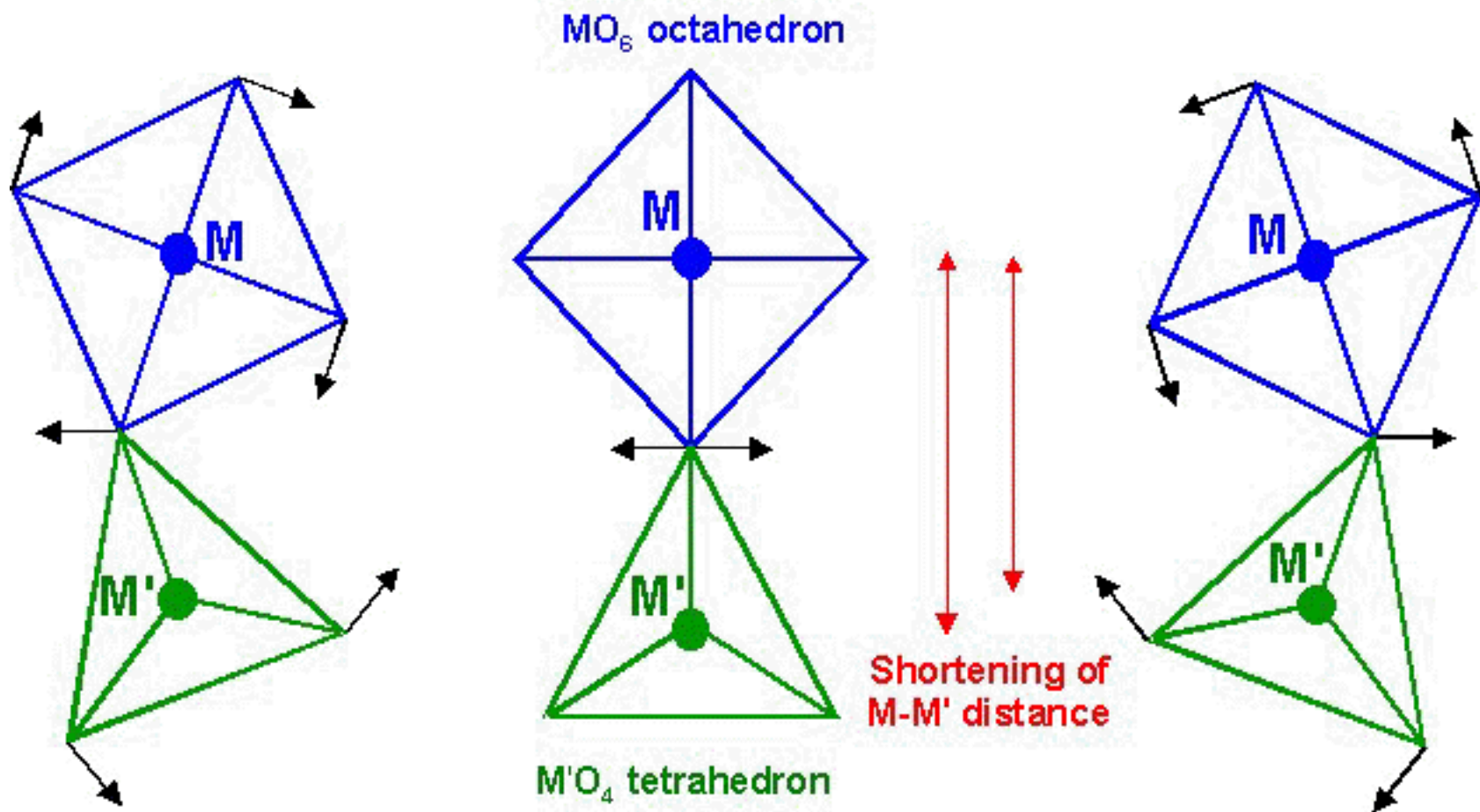
Cubic:

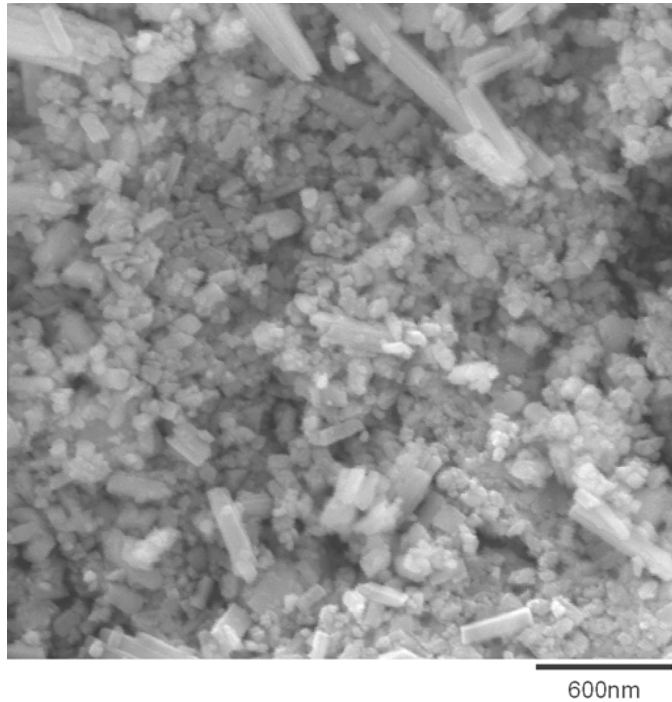


Orthorhombic:

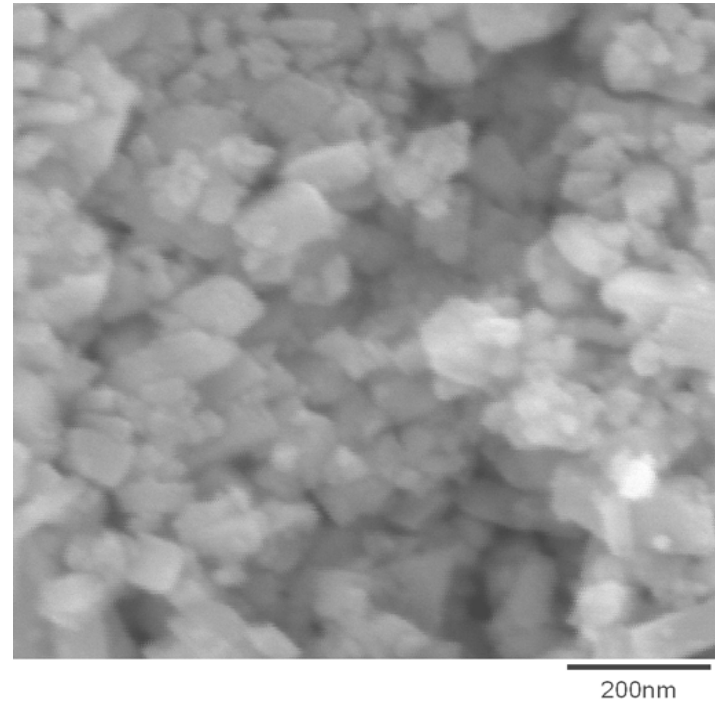


Origin of Negative CTE





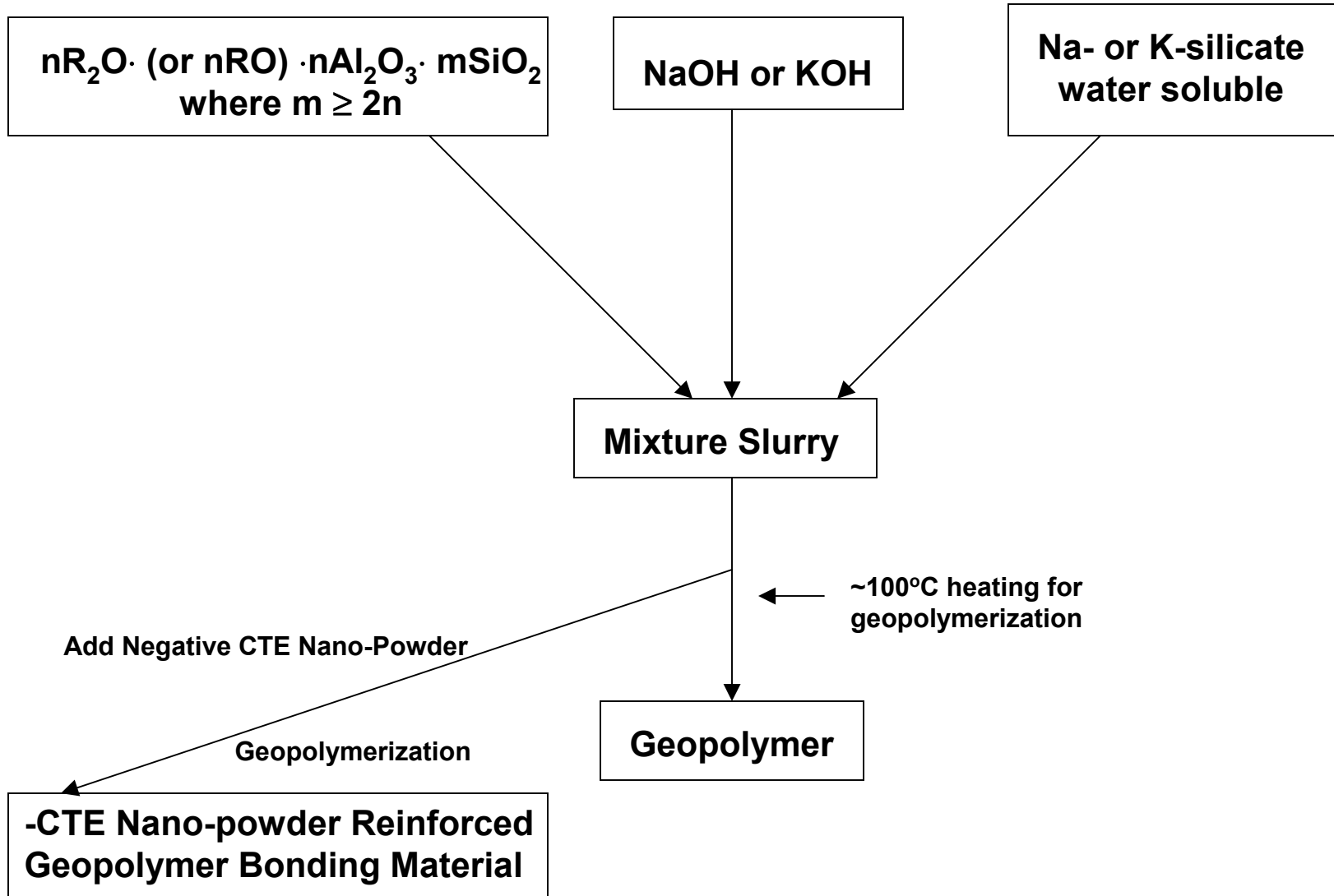
(a)



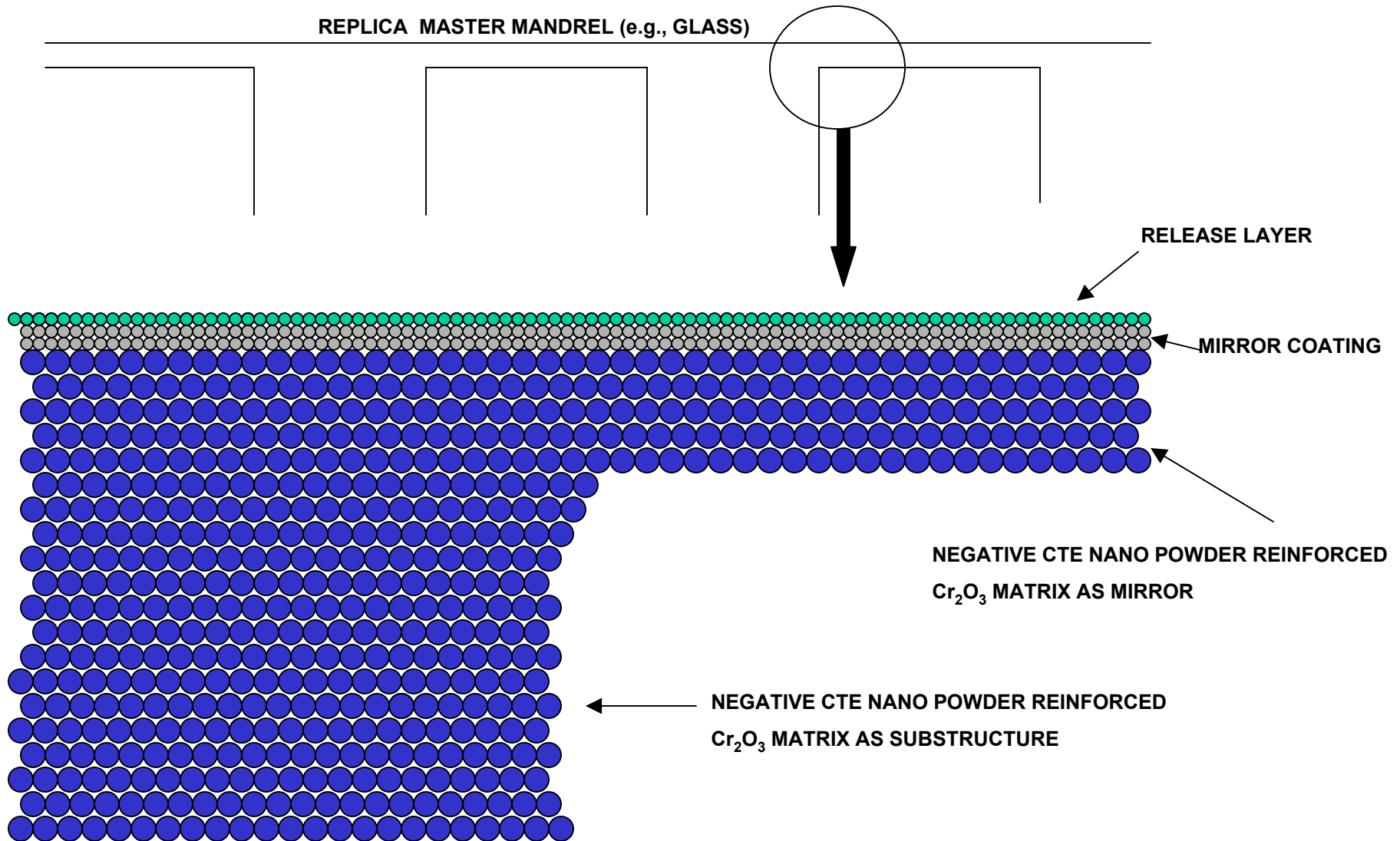
(b)

**SEM Photomicrographs of ZrW₂O₈ Powder (UES)
(a) After Dehydration, and (b) After Calcination**

Geopolymerization

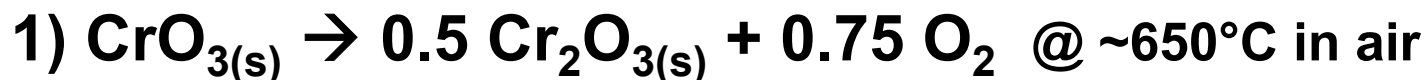


Schematic Drawing of - CTE Nano-Powder Reinforced CDD Processed Uni-Body Composite Mirror Assembly Concept

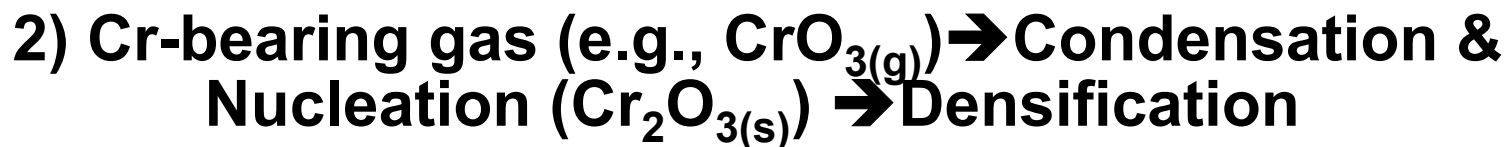


Chemically Driven Densification (CDD) process

Reaction:



CrO_3 : m.p. = 197°C ; decomposition T. = 250°C



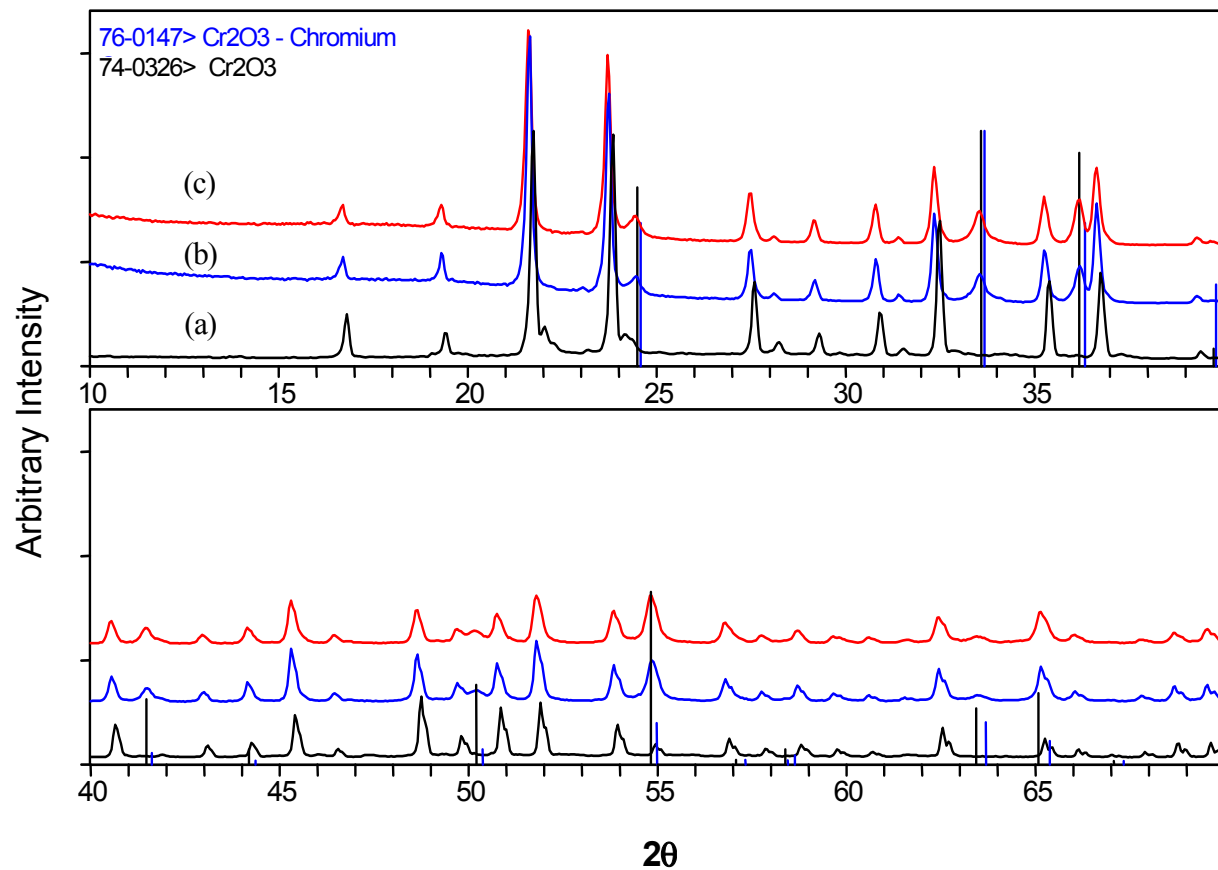
Examples/Experiences:

Densification of Porous Alumina matrix in CMC

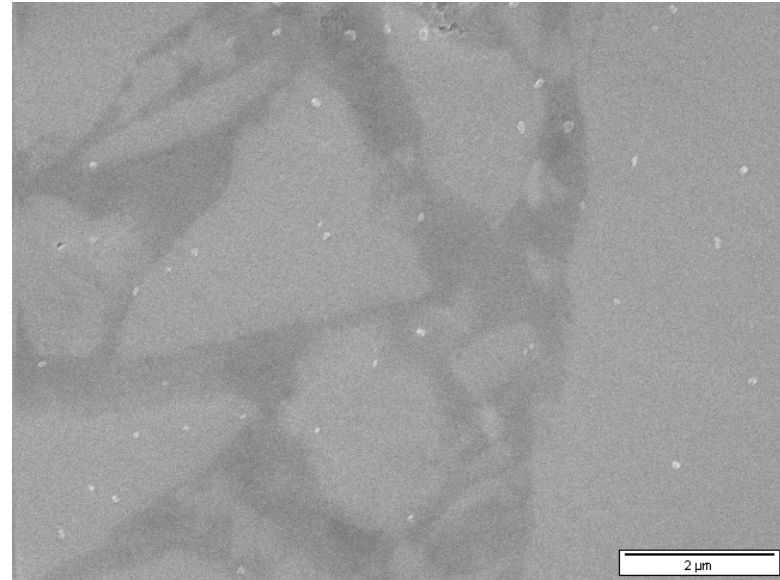
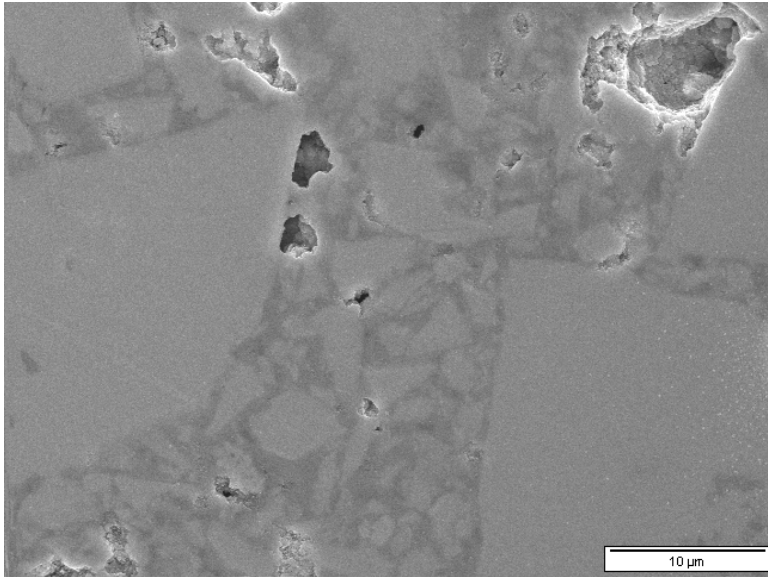
Current Process:

Multiple-Infiltration and Pyrolysis of $\text{CrO}_3/\text{H}_2\text{O}$ Solution into Commercial (Wah Chang) Negative CTE Powder (ZrW_2O_8) Preform

Future Work: Use ZrW_2O_8 Nano-Powder (UES)



**Powder diffraction patterns of (a) ZrW₂O₈,
(b) ZrW₂O₈-Cr₂O₃ (3 cycles), and (c) ZrW₂O₈-Cr₂O₃ (5 cycles)**



SEM Photomicrographs of ZrW_2O_8 – Cr_2O_3 Composite showing insufficient densification due to the presence of large ZrW_2O_8 particles (Wah Chang).

High Magnification shows full densification near fine ZrW_2O_8 particles.

Average grain size of CDD processed Cr_2O_3 matrix is about 25 nm.

(Grey phase = ZrW_2O_8 , Dark phase = Cr_2O_3)

SUMMARY

- * Preliminary Experimental Work on All the Key Processing Issues were Successfully Explored:**
 - Nano-Laminate Deposition**
 - Release Layer Deposition**
 - Negative CTE Nano-Powder Synthesis**
 - Suitable Geopolymer Synthesis**
 - CDD processing with Negative CTE powder**
- * Base on the Preliminary Work, the Proposed Concepts are Appeared to be Viable**

FUTURE WORK

- * Nano-Laminate Deposition Process Optimization**
- * Best Release Layer Selection and Experimentation**
- * Negative CTE Nano-Powder Production in Large Quantity**
- * Bond Strength Measurement between SiC and SiC using Negative Nano Negative CTE powder Reinforced Geopolymer**
- * CDD Processing using Nano-sized Negative CTE Powder Preform and Characterization**
- * Microstructural Characterization and Physical Property (e.g., CTE) Measurement of Nano-Laminate**
- * Based on the Optimization Study, Down Select or Modify the Proposed Concept will be Proposed to Study in for Phase II**